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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

**“पुराने को छोड़ नये के तरफ”**

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12308-3 (1987): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 3: Determination of manganese by periodic spectrophotometric method (for manganese 0.1 to 2.5 percent) [MTD 6: Pig iron and Cast Iron]

**“ज्ञान से एक नये भारत का निर्माण”**

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“Invent a New India Using Knowledge”



**“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”**

Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”





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*Indian Standard*  
**METHODS FOR**  
**CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON**  
**PART 3 DETERMINATION OF MANGANESE BY PERIODATE**  
**SPECTROPHOTOMETRIC METHOD**  
**( FOR MANGANESE 0.1 TO 2.5 PERCENT )**

**1. Scope** — This standard (Part 3) covers the method for determination of manganese in cast iron and pig iron in the range of 0.1 to 2.5 percent.

**2. Sampling** — The sample shall be drawn and prepared as described in the relevant Indian Standard.

**3. Quality of Reagent** — Unless specified otherwise, analytical grade reagents and distilled water [see IS : 1070-1977 Specification for water for general laboratory use (*second revision*)] shall be employed in the test.

**4. Determination of Manganese in Cast Iron and Pig Iron by Periodate Spectrophotometric Method**

**4.1 Outline of the Method** — After dissolution of the sample in sulphuric/phosphoric acid mixture and nitric acid, the solution is fumed with perchloric acid. Manganese is oxidized to permanganic acid by potassium periodate. Absorbance of the solution is measured at 545 nm.

**4.2 Reagents**

**4.2.1 Sulphuric-phosphoric acid mixture** — Add 100 ml of concentrated sulphuric acid (rd 1.84) and 150 ml of phosphoric acid (rd 1.75) to 600 ml of water. Cool and dilute to 1 litre with water.

**4.2.2 Concentrated nitric acid** — (rd 1.42) [Conforming to IS : 264-1976 Specification for nitric acid (*second revision*)].

**4.2.3 Perchloric acid** — 70 percent.

**4.2.4 Dilute perchloric acid** — 1 : 99 (v/v).

**4.2.5 Potassium periodate solution** — Dissolve 7.5 g of potassium periodate in 200 ml of dilute nitric acid (1 : 1) and add 400 ml of phosphoric acid; cool, and dilute to 1 litre.

**4.2.6 Standard manganese solution (1 ml = 0.01 mg Mn)** — Prepare as described in Method A and B:

*Method A (Preparation from manganese metal)* — Take 0.1 g of manganese metal (purity 99.8 percent, Min) in a beaker and add 10 ml of dilute nitric acid (1 : 1). Heat gently until dissolution is complete and brown fumes are expelled. Cool, transfer to 1 litre volumetric flask, dilute to volume and mix. Take 10 ml of the solution and dilute to 100 ml.

*Method B (Preparation from potassium permanganate)* — Dissolve 3.2 g of potassium permanganate ( $KMnO_4$ ) in 1 litre of water. Let stand in the dark for 2 weeks. Filter without washing through a Gooch crucible. Avoid contact with rubber or other organic material. Store in a dark coloured glass stoppered bottle.

**4.2.6.1 Standardization for method B** — Dry a portion of sodium oxalate at 105°C. Transfer 0.300 0 g of sodium oxalate to 500 ml beaker. Add 250 ml of dilute sulphuric acid (1 : 19), previously boiled for 10 to 15 minutes and then cooled to  $27 \pm 3^\circ C$  and stir until the oxalate has dissolved. Add about 40 ml of potassium permanganate solution at a rate of 25 to 35 ml/min, while stirring slowly. Let stand until the pink colour disappears. Heat to 55 to 60°C and complete the titration by adding potassium permanganate solution until a faint pink colour persists for 30 seconds. Find the normality of the solution and adjust to 0.100 0 N.

Transfer 90.9 ml of 0.100 0 N potassium permanganate solution to 500-ml beaker and add 10 ml of dilute sulphuric acid (1 : 1). Reduce the potassium permanganate solution by sulphurous acid and boil the solution until free of sulphur dioxide. Cool, and transfer to 1 litre volumetric flask and make up. Take 10 ml of the solution and dilute to 100 ml.

**4.3 Procedure**

**4.3.1** Take 1·00 g of sample in a 250-ml conical flask, add 50 ml of sulphuric-phosphoric acid mixture. Heat gently until action ceases. When sample is dissolved, oxidize with a few drops of concentrated nitric acid and add 10 ml of perchloric acid ( see Note ). Evaporate until white perchloric acid fumes are given off and keep at this temperature for 10 minutes.

**Note** — If the sample does not dissolve readily in acid mixture, add 5 ml each of concentrated hydrochloric acid and concentrated nitric acid to facilitate the dissolution.

**4.3.2** After cooling, dilute to 60 to 70 ml with water and boil. If necessary; filter and wash with hot dilute perchloric acid. Collect the filtrate in 100-ml volumetric flask. Dilute to mark and mix well.

**4.3.3** Take a suitable aliquot from the above solution (containing 0·1 to 1 mg of manganese) in a 250-ml conical flask and bring it to boil. Add 5 ml of acid mixture and 10 ml of potassium periodate solution and boil at 90°C for 10 minutes. Cool to ambient temperature. Transfer to 10-ml volumetric flask and dilute to mark with water ( see Note ) and mix. Measure the absorbance at 545 nm against a reagent blank.

**Note** — All water used for dilution should be pretreated with potassium periodate.

**4.3.4 Blank** — Carry out a blank using the same quantity of the reagents used.

**4.3.5 Calibration curve** — Transfer 0, 1·0, 2·0, 4·0, 6·0, 8·0 and 10·0 ml of standard manganese solution ( 1 ml = 0·01 mg Mn ) to seven 100-ml volumetric flasks and proceed according to 4.3.3. Draw a calibration curve of absorbance values against milligrams of manganese in the various aliquots.

**4.3.6 Calculation** — Convert the spectrophotometric reading of the sample taken under 4.3.3 to milligrams of manganese by means of calibration curve and calculate the percentage of manganese as follows:

$$\text{Manganese, percent} = \frac{A}{B} \times 0\cdot1$$

where

A = mass in mg of manganese found in the aliquot of the solution, and

B = mass in g of sample represented by aliquot of the solution taken.

**4.3.7 Reproducibility** —  $\pm 0\cdot01$  percent for manganese content up to 0·3 percent,  
 $\pm 0\cdot02$  percent for manganese content between 0·3 to 0·7 percent,  
 $\pm 0\cdot06$  percent for manganese content between 0·7 to 1 percent,  
 $\pm 0\cdot08$  percent for manganese content between 1 to 1·6 percent, and  
 $\pm 0\cdot04$  percent for manganese content between 1·6 to 2·5 percent.

### EXPLANATORY NOTE

Method for determination of manganese in cast iron and pig iron by bismuthate method and persulphate oxidation method were given in IS : 228-1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels ( revised )'. IS : 228-1959 was again revised to make it comprehensive in respect of steel analysis. To cover chemical analysis of cast iron and pig iron, it was decided that a separate standard be published in series. This standard is one of the series of Indian Standards on methods of chemical analysis of cast iron and pig iron, determination of manganese by periodate spectrophotometric method. With the publication of this standard, the bismuthate method will be superseded.

The other parts of the standard are:

Part 1 Determination of total carbon by thermal conductivity method

Part 2 Determination of sulphur by iodimetric titration after combustion.